

ART. XIII.—*Experiments made on a Sample of Pig Iron received from the British and Tasmanian Iron Company, Port Lempriere, Tasmania.*

BY J. COSMO NEWBERY AND FREDERIC DUNN.

[Read 12th December, 1878.]

DURING the month of November, 1876, a sample of pig iron was sent to the laboratory for examination and report. Upon treating a portion of this iron (which had been very finely ground) in a flask and boiling on the sand-bath with nitrohydrochloric acid ($1\frac{1}{2}$ parts of hydrochloric to 1 of nitric acid) the iron was readily attacked. When all action had ceased the supernatant liquor was carefully decanted off from the residue. The latter was found to have a peculiar bronze-like appearance. This powder was at first believed to be "nitride of titanium," but upon further investigation was found to be a compound of chromium iron and carbonaceous matter.

The pig iron which was found to contain the most chromium was coarse-grained and crystalline, having a white lustre somewhat resembling "spiegeleisen" in appearance, but its lustre was of a less brilliant white colour, and the crystal plates very rough.

This sample gave a residue on treatment with nitrohydrochloric acid of 9.38 per cent. of a bronze-coloured chromium compound (calculated to the total pig), whereas a sample of pig iron which was of much finer grain and granular in structure gave 1.52 per cent. of the same peculiar compound.

The pig iron when treated with hydrochloric and sulphuric acids gave different results to that obtained by nitric acid.

TREATMENT WITH HYDROCHLORIC ACID.

The pig iron was broken up into pieces about the size of a bean, placed in a flask, and boiled with hydrochloric acid. After all effervescence had ceased the vessel was taken off the sand-bath and transferred to a quiet place, in order that the small particles might settle at the bottom. The supernatant liquor was then decanted off, the residue was re-treated

with hydrochloric acid, decanted again, and residue well washed and dried. A magnet was then passed through it so as to take up any particles of metallic iron which might be left undecomposed. Upon examining the residue with the microscope, peculiar bronze-coloured, star-like crystals were observed. Owing to the large amounts of silica and carbonaceous matter which are left, it is very difficult to separate the little bronze-like stars. These stellate forms contain a large percentage of chromium as a component part in combination with iron. A sufficient quantity has not yet been obtained for a quantitative analysis. This difficulty is due to their solubility in boiling hydrochloric acid.

If they are boiled with nitric acid they lose their bronzy appearance, and become silvery white; are very slowly dissolved by this acid.

They are very slowly acted upon by sulphuric acid.

TREATMENT WITH NITRIC ACID.

Small pieces of pig iron, if boiled with nitric acid, leave silvery white plates. When these appear the acid solution was carefully decanted off and the plates well washed with distilled water, and re-treated with nitric acid, and boiled. They were washed out into a suitable vessel and dried. These plates are not magnetic, so that any undissolved iron could be removed by a magnet.

A large proportion of these metallic silvery-looking plates are dissolved, owing to their long-continued boiling in this acid.

The following are the analyses which have been made of various samples of this compound:—

	(1)	(2)	(3)	(4)	(5)	(6)
Percentage of iron ...	87.44	83.92	84.78	84.60	84.69	84.44
„ chromium ...	12.71	16.07	15.73	15.40	15.90	15.56
„ carbon ...	trace	—	—	—	—	—
	100.15	99.99	100.51	100.00	100.59	100.00

No. 1. Is the analysis of the first sample of silvery white plates obtained. The plates were not thoroughly freed from undissolved iron, hence the high percentage.

No. 2. This sample was re-treated for some time in nitric acid, washed well with distilled water, dried, and the magnet passed through the mass, and is therefore the purest sample. The iron and chromium were estimated by a process founded on that given by “Crooke’s Select Methods in Chemical Analysis.”

Nos. 1, 3, 5. The chromium and iron were estimated in these samples by the fusion method, which is described in *Fresenius' Quantitative Chemical Analysis*.

Nos. 4, 6. The iron in these samples was carefully determined by a standard solution of permanganate of potash, and the chromium estimated by loss.

In appearance these non-magnetic scales resemble osmiridium, being of a greyish silvery white, and are brittle.

Hydrochloric acid readily dissolves these plates, forming an emerald green solution. Long boiling is required, however, to get a complete solution.

Towards the end of the operation, small particles having a bronze-like appearance float in the liquid; these can only be dissolved by continued boiling in the concentrated acid.

Sulphuric acid readily attacks the plates. They are not acted upon by acetic acid.

A portion of these plates were boiled in a flask with nitric acid for a very long time, and were entirely dissolved.

There is not the slightest doubt that a large percentage of these silvery plates are dissolved, owing to the long-continued boiling which the pig iron receives during its solution in nitric acid.

TREATMENT WITH SULPHURIC ACID.

Stellate forms are obtained if the pig iron be treated in the same manner as is described under the "hydrochloric acid treatment."

TREATMENT WITH NITROHYDROCHLORIC ACID.

A portion of the finely pulverised iron was treated in a flask with hot nitrohydrochloric acid until a bronze-like powder made its appearance; water was then added to stop the action of the acid, and the powder separated and collected; the iron residue was again treated with acid.

The bronze powder thus obtained was purified by re-treating with nitrohydrochloric acid and well washing.

If the bright bronze powder be left exposed to moist air it becomes slightly tarnished and shows a beautiful iridescence.

If boiled in nitric acid for a short time, it loses its peculiar bronzy appearance and is converted into those silvery white non-magnetic scales, the same as those obtained in the residue, after boiling the pig iron in nitric acid.

The filtrate from these plates was tested to see if any chromium had gone into solution; only a slight reaction was obtained.

The bronze powder upon treating with sulphuric acid and boiling is readily attacked, carbonaceous particles being liberated; the continued action of the sulphuric acid on the latter causes the evolution of foetid hydrogen, the solution assuming a brownish black appearance, which upon further boiling assumes a green colour.

Hydrochloric acid dissolves this powder, but the peculiar bronzy appearance remains to the last. The solution is of a fine emerald green colour; carbonaceous particles separate during the solution; a peculiar hydrocarbon smell is evolved.

Acetic acid fails to dissolve this bronze powder, and is therefore useful in separating any free iron which may be mechanically mixed with it.

A portion of the powder was ground in an agate mortar with water; it loses its bronze-like appearance, becoming steel-grey, carbonaceous matter being liberated (this shows that the carbonaceous matter is merely mechanically disseminated), the non-magnetic, metallic particles being left behind.

Upon analysis the bronze powder was found to contain in 100 parts:—

Percentage of iron	81.12
„ chromium	15.09
„ carbonaceous matter	4.11
„ silica	0.53
				100.85

The analysis shows that when separated from the carbon it has the same composition as the nitric acid residue. The following is its composition, after deducting the carbon and silica:—

Percentage of iron	84.32
„ chromium	15.68
				100.00

TREATMENT WITH ACETIC ACID.

100 grains of finely-ground pig iron were placed in a flask and gently boiled with acetic acid.

During solution the acid at first readily attacks the iron, the liquid assuming a green tint, afterwards passing into a deep brown.

After treating the finely-divided iron two or three times with fresh portions of acetic acid, the liquid becomes nearly colourless, holding very little iron in solution, and not any chromium; on further boiling with acetic acid chromium was taken into solution.

When all action had ceased there were obtained 37 grains of insoluble pig iron, of which 2.20 grains were non-magnetic, metallic particles, the remaining 34.8 grains being magnetic.

Upon analysis the non-magnetic portion gave in 100 parts:—

Percentage of iron	85.39
„ chromium	14.90
„ carbon	trace
				<hr/>
				100.29

On treating these particles with nitric acid they are converted into those silvery white plates. They correspond exactly to the non-magnetic particles mentioned under the heading of “Treatment with the Magnet.”

Upon treating a portion of the magnetic particles in boiling hydrochloric acid, a few bronze-like stars were obtained, corresponding to those mentioned under the “Hydrochloric acid treatment;” treating a portion also in boiling nitric acid for a short time the silvery white plates are obtained. These, upon analysis, gave in one hundred parts:—

Percentage of iron	84.60
„ chromium	15.40
				<hr/>
				100.00

TREATMENT WITH THE MAGNET.

The pig iron was ground to a very fine powder. One hundred grains were then placed upon a glazed sheet of paper and the magnet held in close proximity to the mass, when the magnet became covered with metallic particles. These were shaken on to a sheet of paper, thus separating the magnetic from the non-magnetic. The magnetic particles were then ground to a finer state of division, and re-treated with the magnet several times.

By this treatment there were obtained 2·48 per cent. of bright, metallic, non-magnetic particles, and 7·43 per cent. of slightly magnetic, metallic particles.

Upon analysis one hundred parts of the non-magnetic particles contained :—

Percentage of iron	85·44
„ chromium	14·95
„ carbon	trace
				100·39

Upon boiling a portion of these particles in nitric acid, they were converted into the silvery plates.

One hundred parts of the slightly-magnetic particles gave, upon analysis :—

Percentage of iron	87·55
„ chromium	11·28
„ silica and undetermined	1·17
				100·00

A number of these slightly magnetic particles were boiled in a flask with nitric acid (1 part of nitric acid with 2 parts of distilled water) until the solution ceased to be coloured by the dissolved iron. Those silvery white particles as mentioned under the “Nitric Acid Treatment,” were obtained.

Upon analysis these plates gave in 100 parts :—

Percentage of iron	84·44
„ chromium	15·56
				100·00

This shows that a large percentage of those non-magnetic silvery plates are left in the magnetic mass, even after very careful treatment with the magnet; this no doubt is owing to the plates being impregnated with the surrounding particles of metallic iron.

The quantity of star-like forms in the hydrochloric and residue did not suffice for an exact analysis, but their behaviour with acids shows that they differ in composition from the silvery plates.

One sample of the iron gave minute prismatic needles in place of plates, upon treatment with nitrohydrochloric acid.

These examinations show that the assumption that the chromium is alloyed or combined with the whole mass of

the iron is incorrect, but that at any rate, most, if not the whole, of it is as two or more definite compounds of iron and chromium diffused through the mass of iron. Different portions of the same pig iron contain variable percentages of these compounds.

The sample of pig iron from which these results were obtained gave 8.98 per cent. of chromium in one part, and 6.63 per cent. in another.

ART. XIV.—*Formation of Hyalite by the Action of Ammonia.*

BY J. COSMO NEWBERY, B.Sc.

[Read 12th December, 1878.]

IN the examination of building stones used in Melbourne I have noticed that the greatest amount of decay takes place during the summer months, December, January, and February, and that the stones which harden on exposure harden most during those months; also, that taking two portions of the same stone, saturating one part with water, and leaving the other dry, the wet stone hardens first, the hardening taking place from the outside inwards.

Analysis of the outer portions of these hardened stones shows an excess of silica, more or less hydrous, and nearly always giving distinct traces of ammonia.

In the Geological Survey Reports, Nos. 4 and 5, I have called attention to some of these peculiar passages of silica from the inner to the outer parts of the stone, and shown that all our freestones, except those already hardened by exposure, are acted on with considerable rapidity by ammonia and carbonate of ammonia. Some are hardened by this action and some are disintegrated. Those which are destroyed fall gradually away, the cementing material being decomposed by the ammonia, and the quartz grains are left free to fall or be washed away by the rain.

In the stones which are not destroyed but harden, some other action takes place; the cementing material between the sand grains is not softened, but it changes from a dull